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MATERIALS RESEARCH FOR THE CLEAN UTILIZATION OF COAL

Quarterly Progress Report

July - September 1981

S. J. Schneider
Project Manager

Center for Materials Science
National Bureau of Standards
U. S. Department of Commerce
Washington, D. C. 20234

PREPARED FOR THE UNITED STATES DEPARTMENT OF ENERGY

Office of Advanced Research and Technology

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I. SUMMARY OF PROGRESS TO DATE

Brief Summary

1. Materials Performance and Properties (H. M. Ondik, B. W. Christ, A. Perloff, and W. A. Willard)

The major effort this quarter has continued to be concentrated on finalizing the first edition of the book, "Construction Materials for Coal Conversion--Performance and Properties Data". The "Materials Evaluation" sections are nearing completion, and the "Index" section is being keyboarded for computerized manipulation and printing. Preparation of next year's plans and a talk to be given at the Sixth Annual Conference on Materials for Coal Conversion and Utilization was done. Regular activities of cataloging and abstracting reports and responding to queries are continuing.

2. Creep and Related Properties of Refractories (N. J. Tighe, C. L. McDaniel, S. M. Wiederhorn)

Work during the quarter was concentrated on obtaining static load data for hot-pressed silicon carbide and alpha alumina. Five compositions of fused cast oxide refractories were obtained from the Refractories Division of Carborundum for use in our testing program. These are 6 x 4 x 24 in and are the smallest size available. The 5 compositions and product designations are:

1.	alumina-zirconia-silica	Monofrax CS-3
2.	alpha-beta alumina	Monofrax M
3.	alpha alumina	Monofrax A
4.	chrome-alumina	Monofrax K-3
5.	chrome-alumina spinel	Monofrax E

1. Materials Performance and Properties (H. M. Ondik, B. W. Christ, A. Perloff, and W. A. Willard)

Progress:

Data Center activities for the past quarter have included the regular data activities; receiving and cataloging reports, answering queries, abstracting data, etc., as well as work on the book, "Construction Materials for Coal Conversion--Performance and Properties Data". Time during the quarter was also spent in preparation of plans for the next year of the project and in gathering material for a talk to be presented at the Sixth Annual Conference on Materials for Coal Conversion and Utilization in October at NBS. The topic of the talk is "Information Systems for Fossil Energy Materials Applications" and will present an overview of data bases which are of use in the fossil energy field, as well as a description of the data bases operated by this Data Center.

In preparation for setting up the full computer data base for Materials Properties beginning in the next quarter, the bibliography has been coded for computer input so that keyboarding of the information may start with the next contract period.

Completion of the first edition of the book of data for construction materials has been the main occupation of the Data Center staff for this last quarter. The "Performance Data" portions of Section A have proven more time-consuming to prepare than was at first anticipated, but they are being put into final manuscript form. These "Materials Evaluations" pages will include summaries and evaluations of the data which appear in Section B, "Materials Testing Results". The index, which will enable users to find data for specific materials and specific properties, is so vital to the usefulness of the book that great care must be taken in its preparation. A written form of the index has been completed and is being keyboarded for entry into computer files. Since the index cannot be considered finished unless all portions of the book are included, it is necessary to make provision for entry of index items as sections of the book are completed in manuscript form. With the portion of the index which is finished so far in the computer, it will be possible to add items, sort, edit, and even prepare the manuscript version of the index under computer control. It is anticipated that the draft manuscript of the first issue of the book will be delivered to the Department of Energy program monitor by the end of October.

Plans:

The manuscript of the book, "Construction Materials for Coal Conversion--Performance and Properties Data", will be delivered to the program monitor at DoE. The major keyboarding effort will be started to place the data of the Materials Properties Data Base on magnetic tape and into the computer data base management system. The routine activities of the Data Center, with respect to receipt and handling of reports and data and the response to queries, will continue.

2. Creep and Related Properties of Refractories (N. J. Tighe, C. L. McDaniel, S. M. Wiederhorn)

Experimental Procedure: Static load data were obtained on, 1) alpha alumina (Monofrax A) bars 5 x 10 x 50 mm at 1200 °C, and 2) hot-pressed silicon carbide (NC 203) bars 3 x 4 x 50 mm at 1200 °C, 1400 °C and 1500 °C. The bars were tested in four point bend configuration and the specimen deflections were measured continuously during the test periods of up to 1000 hours.

Results:

Alpha alumina (Monofrax A): The static load tests for the fused-cast bar specimens were scheduled to last 1000 hours with a stress of 20 MPa in order to compare the results with the simple compression tests. The first test of 3 specimens terminated at 596 hours because of a cooling water failure; the specimens were cooled to room temperature under load. The deflection curve, shown in figure 1, was obtained from the LVDT readings and was plotted for one hour intervals for the first 24 hours and at 24 hour intervals for the remaining 23 days. As seen in the curve a constant deflection rate of $5.6 \times 10^{-9} s^{-1}$ was recorded after ~50 hours under load. The measured deflection rates for the two regions of constant deflection are shown in Table 1.

Table 1. Static Load Deflection Rate for Fused Cast Alpha Alumina at 1200 °C and 20 MPa.

Specimen	Region I (in/in/s)	Region II (in/in/s)
1	8.1×10^{-8}	1.1×10^{-8}
2	4.4×10^{-8}	4.7×10^{-9}
3	6.1×10^{-8}	5.6×10^{-9}

The test bars were bent noticeably when removed from the furnace but had not fractured during cooling. Only alpha alumina was present in the x-ray diffraction pattern in accord with earlier observation on the loss of beta alumina during heating. A second series of samples is being run to complete the 1000 hour test.

It should be noted that the Monofrax A material contains 3-10% beta alumina in the as-received condition and that the beta phase is lost after heating at 1200 °C. This behavior was discussed in earlier reports.

Silicon Carbide (NC 203): Silicon carbide has important uses in high temperature moderate stress applications. Our initial strength measurements were made after exposure at 1200 °C, 1400 °C and 1500 °C for times up to 1000 hours. The results of the tests for averages of 3 specimens per furnace are shown in Table 2. It is clear that the strength decreases significantly after exposure at 1500 °C.

Table 2. High Temperature Flexure Strength of SiC (NC 203)

	Temperature °C	Time, hr.	Flexure Strength, MPa
"o" load			
	1200	1/2	427 ±92
	1200	168	419 ±137
	1400	168	413 ±23
	1500	168	339 ±13
Static Load			
200 MPa	1200	168	496 ±38
150 MPa	1400	168	419 ±26

Static load tests were undertaken as part of the program to evaluate the strength degradation, and to identify failure mechanisms under stress. During this quarter tests have been conducted at 1200 °C, 1400 °C and 1500 °C under static load of 150-300 MPa for one week. Specimens were broken at the end of the test period at temperature without removing the static load. Loads were selected from consideration of the strengths in Table 2. The initial results are summarized in figure 2 which shows the failure times and the static loads. Creep was not detected at 1200 °C under the loads used; and some specimens failed during the test indicating that crack propagation occurred. The reduction in percent of failures by lowering the applied stress indicates we are close to a static fatigue limit at 1200 °C. At 1400 °C three specimens under a stress of 150 MPa survived for one week and showed little or no creep. The three specimens tested at 1500 °C broke within 9 hours. These test series of one week exposures should be finished during the next quarter.

Plans: The static load test series on silicon carbide NC 203 will be continued to achieve at least one week and then 1000 hour durability tests at 1200 and 1400 °C. Specimens of sintered alpha silicon carbide have been prepared and will be scheduled for the long-term tests during the next few quarters. The Monofrax series will be analyzed to compare the compressive and flexure test procedures. We will try to obtain additional samples of the same production run from Montana State. The fused-cast refractories will be cut into slabs and examined for homogeneity before cutting out test samples. The Refractories Division at Carborundum has done some testing on these materials and we will coordinate our tests with theirs.

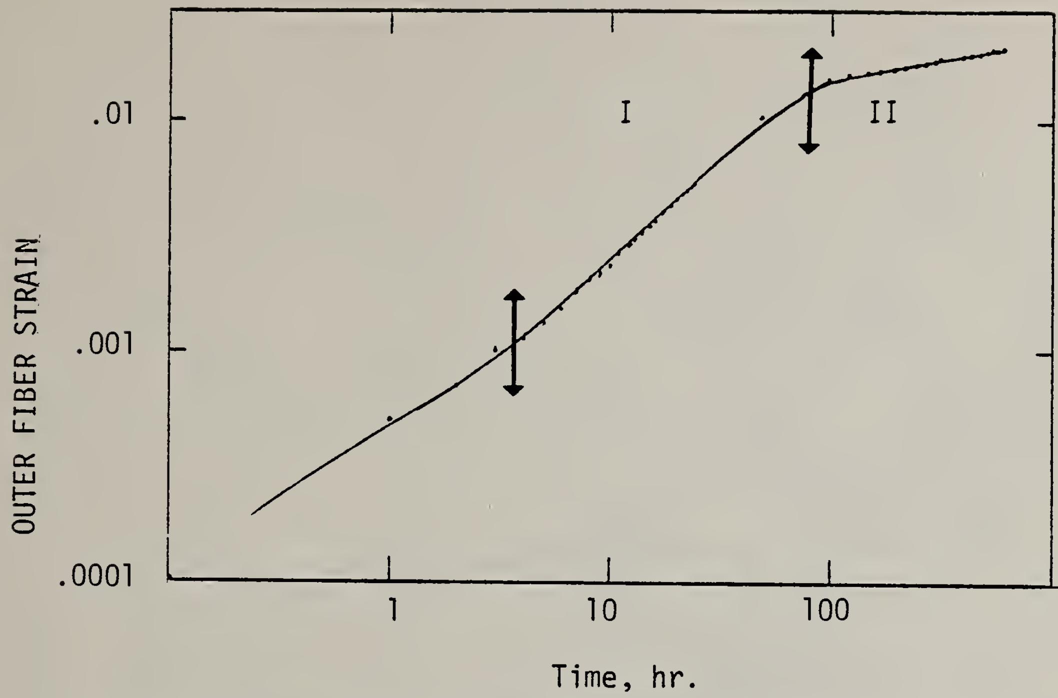

Figure 1. Deflection measured at 1200 °C for alumina (Monofrax A) with static load of 20 MPa.

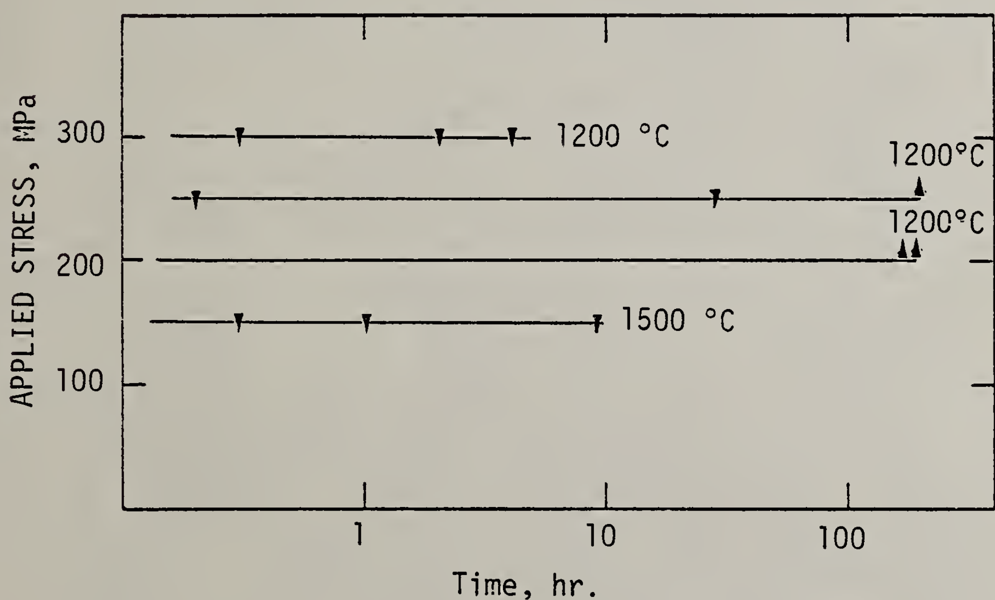

Figure 2. Plot of static load vs. failure time for hot-pressed silicon carbide (NC 203).